IS 5298 : 2013

(Reaffirmed 2018)

भारतीय मानक

आसवन रेंज और आसवन उपज का निर्धारण करने की विधि — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

METHOD FOR DETERMINATION OF DISTILLATION RANGE AND DISTILLATION YIELD

(Second Revision)

ICS 17.200.01

B U R E A U O F I N D I A N S T A N D A R D S
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December 2013 **Price Group 6**

Inorganic Chemicals Sectional Committee, CHD 1

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Distillation range is determined to identify an unknown substance, to measure the approximate purity, or to ascertain whether or not a substance conforms to a specification. In all these cases, reproducibility of results of distillation range depends on close adherence to details, both as regards to apparatus and the procedure. Therefore this standard was first published in 1969, covering an apparatus which was suitable for the determination of distillation range of most of liquid chemicals — both organic and inorganic.

This standard was revised in 1983, on the recommendations of Ministry of Defence, to include provision for ground glass, interchangeable joints, temperature ranges of the water to be circulated, and initial cooling of sample and distillate.

This standard has now been revised to include, procedure for manual distillation apparatus using electric heaters, as an alternate to apparatus using gas burner. Procedure for using automatic distillation apparatus is also included as an alternate to manual distillation procedure. The standard is thoroughly revised to accommodate the above changes.

A separate section on likely hazards involved and precautions to be taken to avoid such hazards has been included in this revision.

For materials, for which the above distillation apparatus are not suitable, the details of appropriate apparatus and procedure would be covered in the individual material specifications.

While reviewing this standard, assistance has also been derived from the following:

ISO 383 : 1976	Laboratory glassware — Interchangeable conical ground joints issued by the International Organization for Standardization
BS 572 : 1985	Interchangeable conical ground glass joints, issued by the British Standards Institution
ASTM D 1078	Distillation range of volatile organic liquids, issued by the American Society for Testing and Materials
ASTM E-133 : 92 (2005)	Standard specification for distillation equipment
ASTM D 86	Standard test method for distillation of petroleum products at atmospheric pressure

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHOD FOR DETERMINATION OF DISTILLATION RANGE AND DISTILLATION YIELD

(Second Revision)

1 SCOPE

This standard prescribes methods for the determination of distillation range and distillation yield of liquids, boiling between 30 and 350°C , that are chemically stable during this process. This method covers manual and automatic distillation procedures. This standard is applicable for both organic and inorganic liquids. This method is not applicable for petroleum products, inorganic acids and alkali solutions. For distillation of petroleum products, IS1448 [P:18] 'Methods of test for petroleum and its products: Part 18 Distillation of petroleum products' is applicable.

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute the provisions of the standard. At the time of publication the additions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of employing the most recent additions of the standards indicated below.

IS No.	*Title*
1448 [P : 18] : 1991	Methods of test for petroleum and its products: Part 18 Distillation of petroleum products
2480 (Part 1) : 1983	Specification for general purpose glass thermometers: Part 1 Solid-stem thermometers
2620 : 1963	Specification for distillation flasks

3 TERMINOLOGY

For the purpose of this standard the following definitions shall apply.

3.1 Distillation — It is the process of vapourizing a liquid and collecting its vapour, which is usually condensed to a liquid. The temperature of the vapour during this process is measured using either a glass thermometer or platinum resistance thermometer. Pure liquids boil at a close boiling range where as liquids containing a homogeneous mixture of different liquids boil in wide boiling range, depending upon the boiling point of individual components.

3.2 Distillation Range — The distillation range of a liquid substance is the range of temperature with in which a specified portion of the liquid distills.

3.3 Distillation Yield — The volume of distillate collected in the receiver, within the corrected, specified temperature range, which is expressed as the percentage by volume of the distillation charge. The specified temperatures to be observed on the thermometer are corrected to standard pressure of 760 mm of Hg, prior to the test if the atmospheric pressure of location where the test is carried out is different from 760 mm of Hg.

3.4 Initial Boiling Point (IBP) —The temperature reading observed at that instant when the first drop of condensate falls from the lower end of the condenser tube into the receiver.

3.5 Final Boiling Point (FBP) — Maximum temperature reading obtained during the test; this usually occurs after the evaporation of all the liquid from the bottom of the flask. The term 'maximum temperature' is a frequently used synonym.

3.6 Dry Point — The thermometer reading which is observed at the instant the last drop of liquid evaporates from the lowest point on the flask. Any drops or films of liquid on the side of the flask or on the thermometer are disregarded.

3.7 Decomposition Point — The thermometer reading that coincides with the first indication of thermal decomposition of the liquid in the flask. It is recognized by the first appearance of white fumes inside the flask.

3.8 End Point 5 min — The thermometer reading obtained 5 min after the 95 percent distillation point, if no dry or final boiling point occurs.

4 PRINCIPLE OF THE METHOD

The sample (100 ml) is distilled under prescribed conditions and the temperature on the thermometer at which specified volume of liquid is collected in the receiver is recorded to evaluate the boiling range of specified volume of liquid, that is recovered by distillation process. Alternatively the initial boiling point and the final boiling point or dry point of the

sample is determined to estimate the total boiling range of a liquid sample. The observed temperatures on the thermometer are corrected to standard barometric pressures. The volume of liquid collected in the receiver between a specified temperature range, or at a particular temperature, which is called the distillation yield can also be determined by this procedure. In automatic distillation platinum resistance thermometer is used instead of mercury in glass type thermometer.

5 SIGNIFICANCE AND USE

This test method provides a method for measurement of distillation range of liquids. The relative volatility of liquid can be used with other tests for identification and measurement of quality. Hence this test method provides a test procedure for assessing quality compliance to a specification. This test method is also useful in manufacturing control where solvents are recovered by distillation process.

6 APPARATUS

6.0 General

All apparatus used for the test shall be thoroughly cleaned and dried.

6.1 Distillation Apparatus — Manual Distillation Method

The details of three different variations of manual distillation apparatus containing draught screen or enclosure, condenser and cooling arrangement are given in Fig. 1 to 3. Figure 1 represents conventional distillation apparatus in which a glass condenser with water circulation arrangement is used for condensing vapour. In Fig. 2 distillation apparatus a metallic condenser immersed in a cooling bath is shown. In both the apparatus a gas burner is used as heat source. Figure 3 represents distillation apparatus in which an electric heater is used as heat source. Figure 4 represents the detailed dimensions of the draught screen (Enclosure) used with manual distillation apparatus shown in Fig.1. Interchangeable ground glass joints, where available may also be used with the apparatus as shown in Fig.1. The details of ground joints are given in Table 1. Further details on the dimensions of various parts of distillation apparatus are given in the subsequent paragraphs.

6.2 Distillation Flask

Distillation flask of 125 ml capacity, made of borosilicate glass complying with dimensions and specifications given in Fig. 5A or Fig. 5B is used in this test method (*see* IS 2620).

6.3 Temperature Measuring Device

6.3.1 *Thermometer*

Total immersion type thermometer of appropriate range shall be selected for manual distillation. The detailed dimensions of solid-stem general purpose glass thermometers are given in Table 2 [*see* IS 2480 (Part 1)].

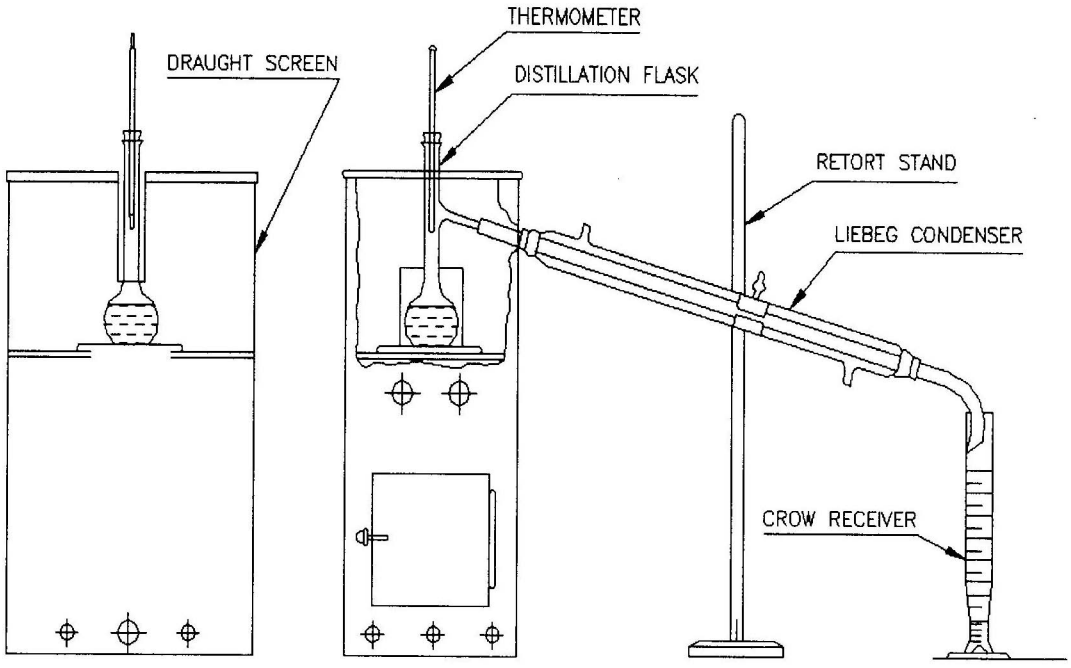


FIG. 1 ASSEMBLY OF MANUAL DISTILLATION APPARATUS USING GLASS CONDENSER

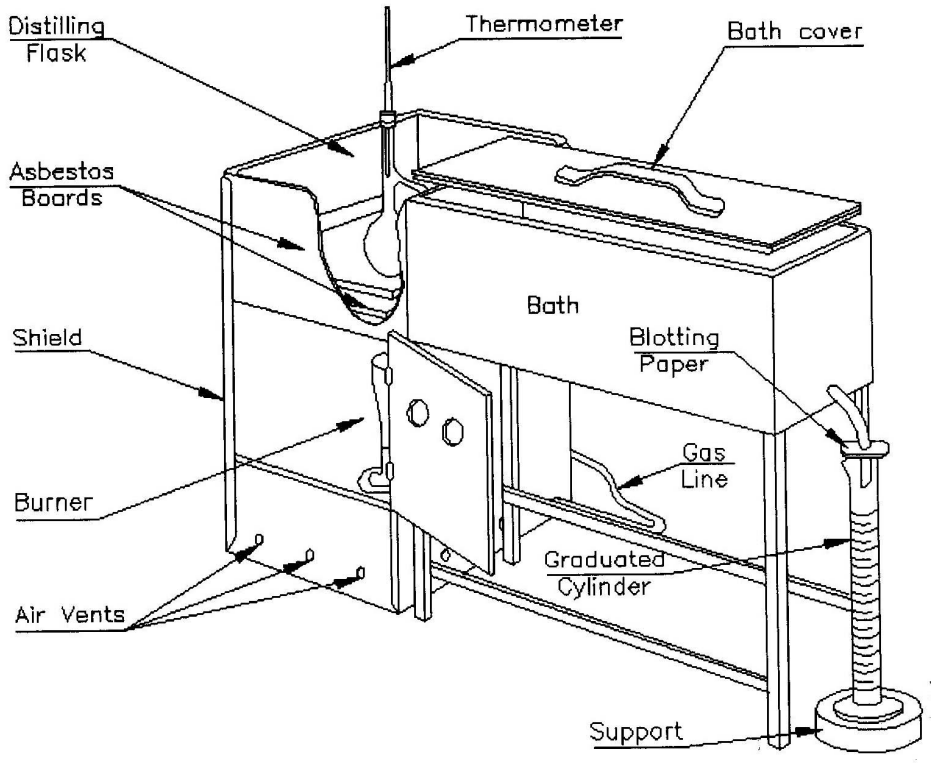


FIG. 2 ASSEMBLY OF MANUAL DISTILLATION APPARATUS USING METALLIC CONDENSER WITH BATH

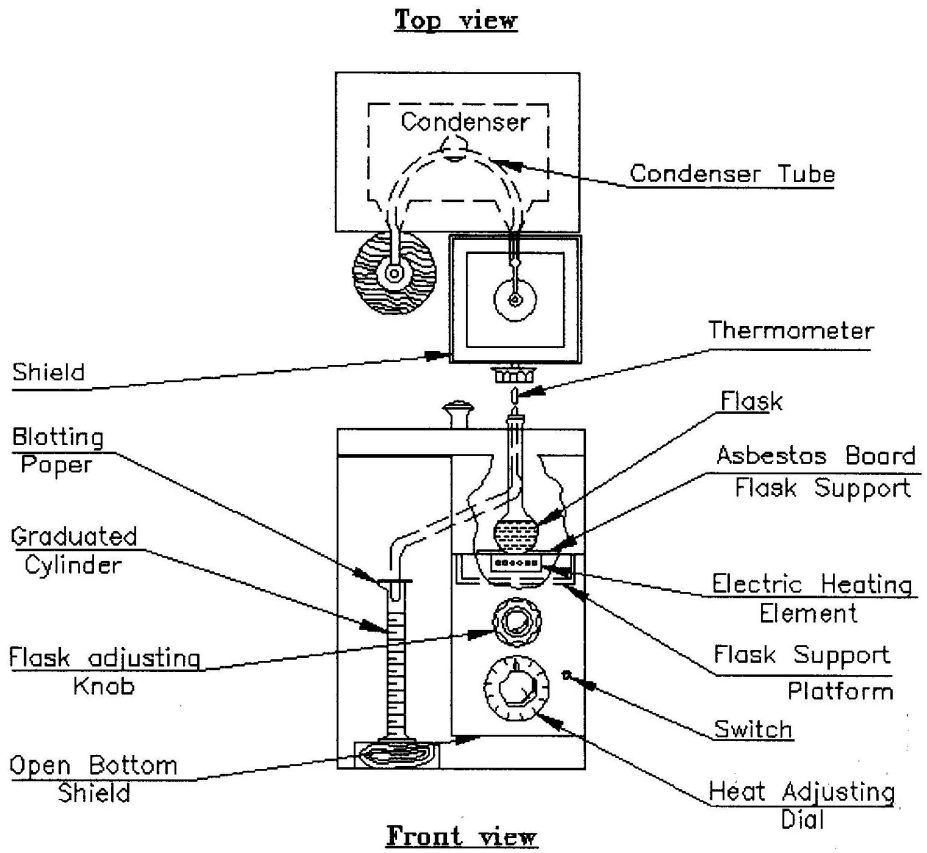
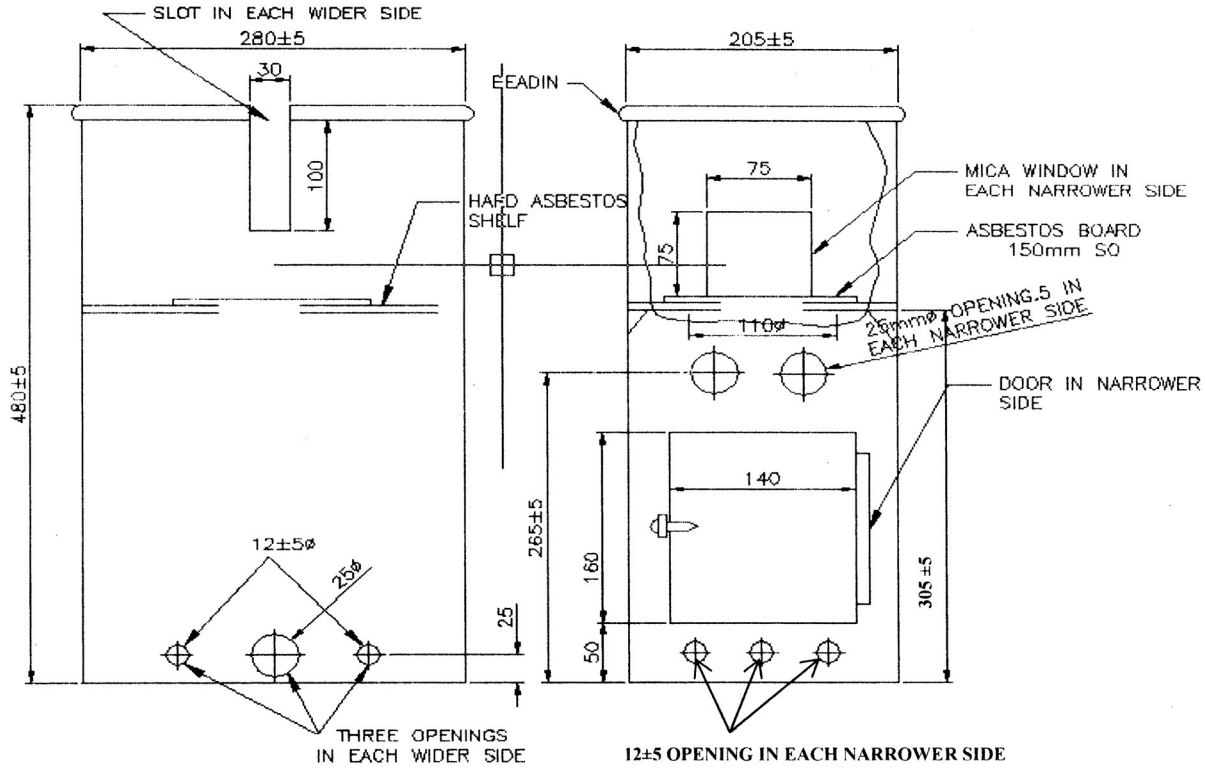
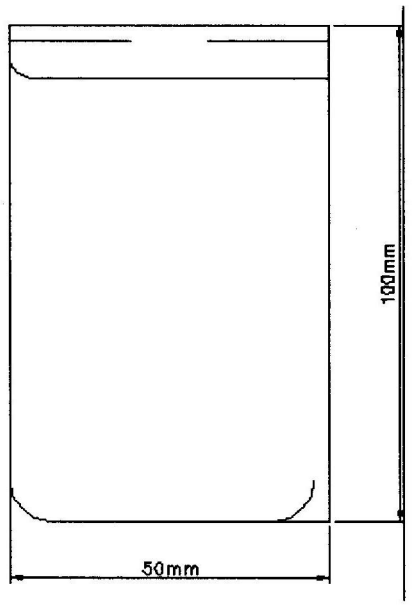


FIG. 3 MANUAL DISTILLATION APPARATUS USING ELECTRIC HEATER AND CONDENSER BATH



4A Cross-Section View of Draught Screen



4B Removable Shutter for the Draught Screen

All dimensions in millimetres.

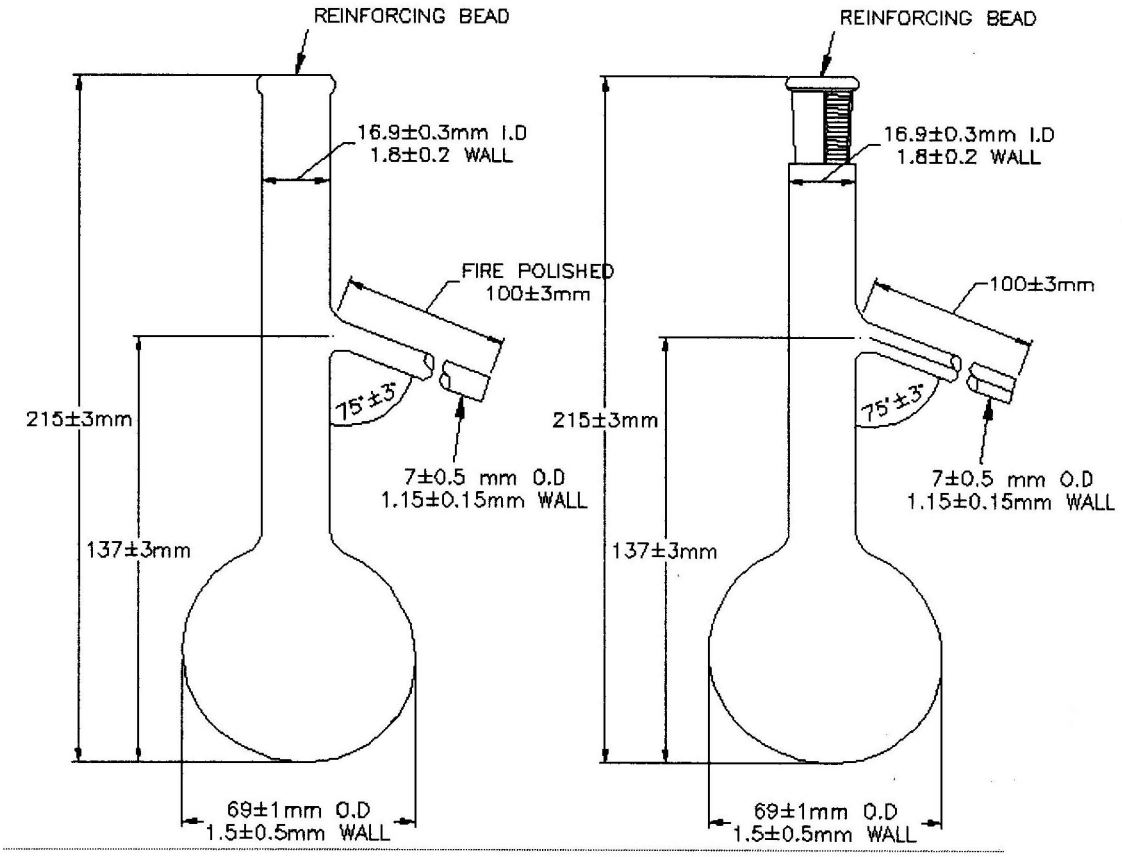
FIG. 4 DRAUGHT SCREEN (ENCLOSURE)

Table 1 Details of Ground Glass Joints

(*Clause* 6.1)

All dimensions in millimetres.

Sl No.	Name of the of Joint	Designation	Diameter of the Ground Zone		Axial Length of the Ground Zone
			Large End	Small End	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Flask to thermometer	B 14	14.5	12.2	23
ii)	Flask to condenser	B 19	18.8	16.2	26
iii)	Condenser to delivery tube	B 19	18.8	16.2	26



5A 125 ml Distillation Flask Without Ground Joint Provision in the Neck

5B 125 ml Distillation Flask with Ground Joint Provision in the Neck

FIG. 5 DISTILLATION FLASKS

For samples having narrow distillation range and for pure liquid chemicals where the total distillation range is less than 2°C, more precise thermometers having smallest division of 0.2°C may be used.

6.3.2 *Automatic Distillation — Temperature Measuring Device*

In automatic distillation systems thermocouples or resistance thermometers are used as temperature measuring device. These thermometers exhibit temperature lag similar to mercury in glass type thermometers. Periodic calibration of these thermometers is accomplished by comparing potentiometrically with the use of standard precision resistances depending on the type of probe. Alternatively pure (99.9 percent) Toluene is distilled

Table 2 Detailed Dimensions of Solid-Stem General Purpose Glass Thermometers
(*Clause* 6.3.1)

Bulb length : 10 to 25 mm
Bulb diameter : Not greater than stem diameter
Stem diameter : 5.5 to 8 mm

Sl No.	Schedule Mark	Nominal Range	Smallest Division	Longer Lines at Each	Fractional Figuring at Each	Full Figuring at Each	Overall Length *Max*	Length of Main Scale *Min*	Distance from Bottom of Bulb to Start of Main Scale *Min*
	TI/PI	°C	°	°C	°C	°C	mm	mm	mm
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	1	–100 to +50	1	5	—	10	305	180	60
ii)	2	– 50 to +60	0.5	1	5	10	305	180	60
iii)	3	– 35 to +50	0.5	1	5	10	305	180	60
iv)	4	– 10 to +110	1	5	10	100	305	180	60
v)	5	– 10 to +110	0.5	1	5	100	305	180	60
vi)	6	– 10 to +160	1	5	10	100	305	180	60
vii)	7	– 10 to + 200	1	5	10	100	305	180	60
viii)	8	– 10 to + 250	1	5	10	100	325	200	60
ix)	9	– 10 to + 360	1	5	10	100	350	225	60
x)	10	– 10 to + 360	2	10	20	100	305	180	60
xi)	11	– 10 to + 500	5	10	50	100	305	180	60
xii)	12	– 0 to + 60	0.25	1	5	10	305	180	60
xiii)	13	– 0 to + 50	0.25	1	5	10	305	180	60

NOTE — TI = Total immersion type thermometer, PI= Partial immersion type thermometer. For Schedule Mark 1 and Mark 2 Partial immersion type not permitted. For the rest thermometers shall be made by increasing the dimensions in col 8 and 10 depending on the immersion required.

using the automatic distillation system and temperature readings are compared with the readings obtained by manual distillation procedure, using mercury in glass type thermometer. The boiling point of Toluene is 110.6°C at 760 mm of Hg pressure.

6.4 Distillation Receiver

6.4.1 *Manual Distillation Receiver*

A 100 ml borosilicate glass cylinder, graduated in 1ml subdivisions and having an overall height of 250 to 260 mm is used for collecting distillate during the test. See Fig. 6A and 6B for detailed dimensions of the distillation receiver.

For measuring the distillation range of a sample below 5 ml volume, distillation receiver shown in Fig. 6A, which is having graduation figures from 1 ml onwards, may be used.

6.4.2 *Automatic Distillation Receiver*

A receiver to be used with automatic distillation is in accordance with the instructions given in the operation manual of the instrument supplier and dimensions shall conform to the values given in Fig. 6B. The automatic distillation receiver will not have visible graduations from 0 to 90 ml. The volume is measured by level follower provided in the automatic distillation apparatus.

6.4.2.1 *Automatic distillation level follower*

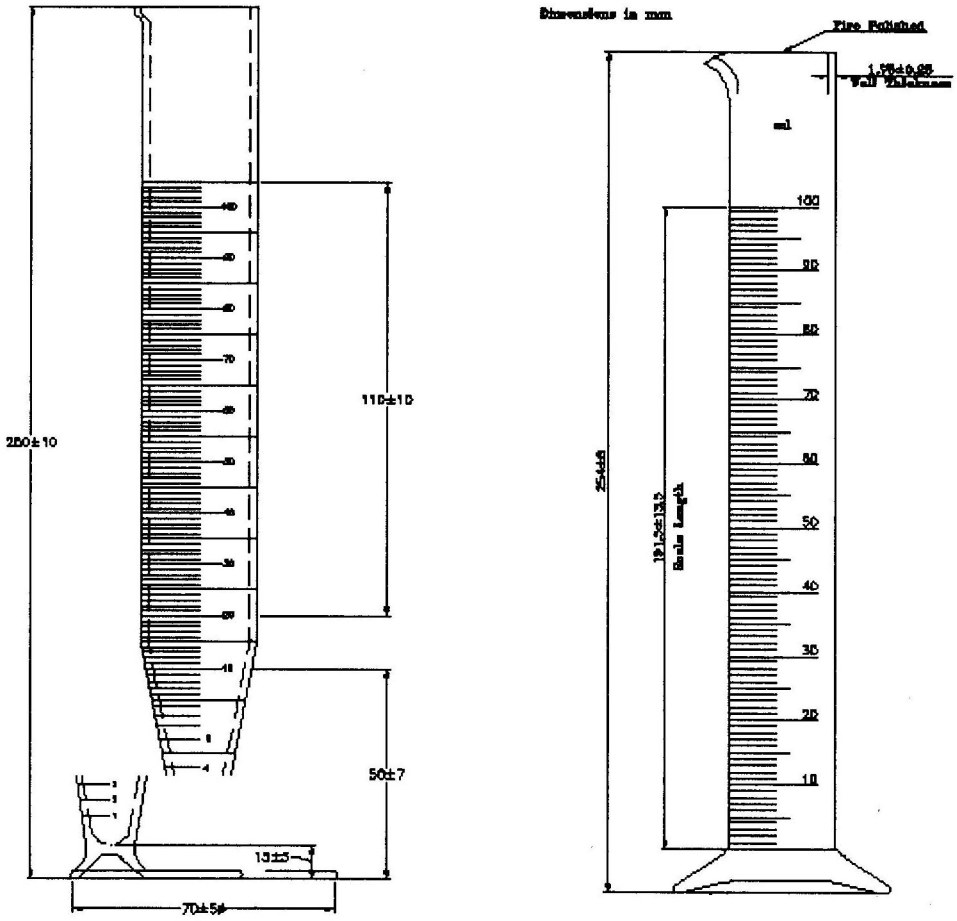
The level follower/recording mechanism of the automatic distillation apparatus shall have a resolution of 0.1ml.

6.5 Condenser

6.5.1 Liebig condenser made of glass with the following dimensions shall be used with distillation apparatus given in Fig. 1 (*see* Fig. 7 for details).

a) Inner tube dimensions : Internal diameter : 14 ± 1.0 mm
Wall thickness : 1.0 - 1.5 mm
Length of shorter limb : 55 ± 5 mm
Length of straight portion of longer limb : 600 ± 10 mm
Angle inclined between shorter and longer limb : 97 ± 3°

b) Outer jacket dimensions : Outer dia of condenser jacket : 35 ± 3 mm
Length : 450 ±10 mm
Water inlet and water out let nozzles shall be provided



6A Receiver with Detailed Graduations Below 5 ml Mark

6B Receiver with Uniform Graduation Marks

FIG. 6 DISTILLATION RECEIVERS

The inlet of the inner tube of the condenser is finished square with its axis, and its outlet is smoothly ground at an angle of approximately 45° with the axis of the tube at that point. The central straight portion of the water jacket is 450 ± 10 mm in length and its external diameter is 35 ± 3 mm.

6.5.2 Cooling bath is used in distillation apparatus shown in Fig. 2 for condensing vapours generated in the distillation process. Metallic condenser made of seamless brass tubing is used in this apparatus. The detailed dimensions of the condenser are given below:

Total length	: 559 mm (22 inch)
Out side dia	: 14.3 mm (9/16 inch)
Wall thickness	: 0.8 - 0.9 mm (0.031 to 0.036 inch)
Length of tube in contact with cooling medium	: 394 mm (15.5 inch)
Tube length out side cooling bath upper end	: 50 mm (2 inch)
Tube length out side cooling bath lower end	: 110 mm (4.5 inch)

The upper end shall be straight with 75 ± 3° to vertical

The lower end of the condenser tube shall be curved downward to a length of 76 mm (3 inch) slightly backward to ensure contact with the wall of the receiver at appoint approximately 25 to 32 mm below the top of the graduated receiver, while carrying out distillation. The lower end of the condenser tube shall be cut-off at an acute angle so that the tip of condenser tube shall be in contact with wall of the receiver. The section of the condenser tube inside the bath is straight in the apparatus shown in Fig. 2 and it is curved in the apparatus shown in Fig. 3. The average gradient of

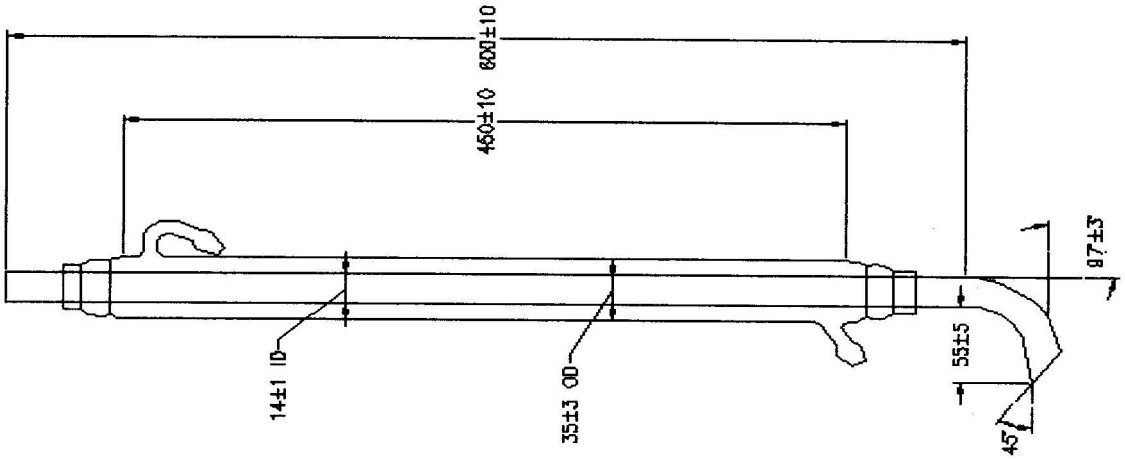


FIG. 7 GLASS CONDENSER USED WITH DISTILLATION APPARATUS GIVEN IN FIG. 1

immersed portion of condenser in side the bath shall be 6.6 mm (0.26 inch).

The capacity of cooling bath shall be not less than 5.5 litre of cooling medium. The arrangement of the tube inside the bath shall be 32 mm below the top edge at the flask side and 19 mm above the floor of the bath towards receiver side. The clearance required between the condenser tube and wall of the bath is 12.7 mm minimum, except near entrance and exit points.

For automatic distillation also the dimensions of condenser bath given in this section are applicable.

6.6 Draught Screen — For Manual Distillation with Gas Burner

The draught screen is rectangular in cross-section and open at top and bottom. It has the dimensions shown in Fig. 4A and 4B and is made of sheet metal of 0.8 mm thick.

6.6.1 In each of the two narrower sides of the draught screen there are two circular air vent holes 25 mm in diameter, situated below the asbestos shelf, as shown in Fig. 4A.

6.6.2 In each of the four sides of the draught screen there are three air vent holes with their centres 25 mm above the base of the draught screen. These holes occupy the positions shown in Fig. 4A. The diameter of each of these holes centrally situated in the wider sides being 25 mm and the remaining 10 holes of 12.5 mm each in diameter.

6.6.3 At the middle of the each of the wider sides, a vertical slot for the side tube of the distillation flask, and dimensions as shown in Fig. 4A is cut downward from the top of the screen. A removable shutter conforming to the dimensions given in Fig. 4B is provided for closing, whichever vertical side not in use.

6.6.4 A shelf of hard asbestos board 6 mm in thickness and possessing a centrally cut circular hole 110 mm in diameter, is supported horizontally in the screen and fits closely to the sides of the screen to ensure that the hot gases from the source of heat do not come in contact with the sides or the neck of the flask. The supports for this asbestos shelf consists of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

6.6.5 In one of the narrower sides of the screen, a door is provided having dimensions shown in Fig. 4A and overlapping an opening in the screen, by approximately 5 mm all around.

6.6.6 In one of the narrower sides of the screen, a mica window is provided centrally with the bottom of the window level on the top of the asbestos shelf. The dimensions and the positions of the window are shown in Fig. 4A.

6.7 Draught Screen (Enclosure) for Manual Distillation with Electric Heater

A typical draught screen for electric heater would be 440 mm high, 200 mm long and 200 mm wide made of sheet metal of 0.8 mm thick, with a window on the front side. An elliptical opening is provided in the rear side of the screen below the top edge for accommodating the upper end of the condenser tube. A flask adjusting knob is provided in the front side of the enclosure for fixing the distillation flask by adjusting the height of the flask support. A heat adjusting indicating knob with dial is provided in the front face of the enclosure for step-less heat control of the electric heater. Other details of the enclosure are similar to draught screen used for gas burner.

6.8 Asbestos Board

In addition to the asbestos shelf referred in **6.6.4** an

asbestos board of 150 mm square and 6 mm thick is required to support the distillation flask. This board contains a circular hole at the centre of the square. The diameter of the hole depends on the final boiling point of the liquids tested for distillation range. For liquids having initial boiling point below 150 °C the diameter of the hole is 32 mm. For liquids boiling above 150 °C the diameter of the hole is 38 mm.

6.9 Source of Heat — Manual Distillation Method

An adjustable gas burner or electric heater, so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in the procedure given in **10** shall be used. For the liquids of narrow boiling range (less than 2°C) an electric heater may be used only if it has been proven to give results comparable to those obtained when using gas burner. An adjustable electric heater with 0 to 1 000 W range has been found satisfactory. For narrow boiling range liquids electric heater may give higher boiling range due to superheating effect of electric heater (*see* Annex A).

6.10 Source of Heat — Automatic Distillation Unit

Electric heaters are used as heat source in automatic distillation units. Follow the manufacturer's recommendations for controlling the heating to maintain the distillation rate with the limits given in the procedure.

7 HAZARDS AND PRECAUTIONS TO BE TAKEN

7.1 Peroxide Formation

Certain solvents and chemical intermediates like ethers and unsaturated compounds may form peroxides during storage. These peroxides may lead to violent explosion hazard when such chemicals are distilled, especially as the dry point is approached. If presence of peroxides in the sample is suspected the material should be analysed and if they are present in sufficient concentration they shall be destroyed before carrying out the distillation test.

7.2 Fire Hazard

Most organic solvents and chemical intermediates burn in the air. In the operation of the distillation apparatus use suitable catch pan and shielding to contain spilled liquid in the event of accidental breakage of the distillation flask during the test. Keep suitable fire extinguishing device near by to put off accidental fire.

7.3 Provide adequate ventilation to maintain solvent vapour concentration below the lower explosive limit in the immediate vicinity of the distillation apparatus and below the threshold limit value in the general work area.

8 PREPARATION AND ASSEMBLY OF APPARATUS

8.1 Manual Distillation Apparatus

8.1.1 Clean and dry the glass condenser tube before starting distillation. If condenser bath is used clean the metallic condenser by swabbing with a piece of soft lint-free cloth attached to a wire or cord, or by any other suitable means.

8.1.2 Use suitable thermometer with smallest graduations that covers the entire distillation range of the product under study. Centre the thermometer into the neck of the flask through a tight fitting cork stopper (which does not react with the test material) so that the upper end of the contraction chamber or mercury bulb of the thermometer is level with the lower side of the vapour tube at its junction at the neck of the flask. Use B14 ground glass joint, if ground glass joint is used in place of cork for fixing the thermometer.

8.1.3 Place the correct flask support (asbestos board) on the shelf provided inside the draught screen. For liquids having initial boiling point below 150°C use flask support with hole size of 32 mm diameter. For liquids with initial boiling point above 150°C use flask support with hole size of 38 mm.

8.1.4 Connect the flask to the condenser by inserting the vapour tube of the flask into the condenser, by making a tight connection with a well rolled cork or of suitable material. If ground glass joint is used, use B19 ground joint for connecting vapour tube to glass condenser. Adjust the position of the flash support and cork on the side tube so that the flask is vertical and the end of the side tube of the flask extends minimum 25 mm in to the condenser and it is coaxial with the condenser. Make sure that the flask bottom is properly rested on the flask support and it completely closes the hole present in the asbestos flask support.

8.1.5 Fill the condenser bath with water or cooling medium. The temperature of the cooling bath shall be maintained as per the details given in Table 3 based on the expected initial boiling point of the material to be distilled.

8.1.6 Cool the sample prior to distillation to the temperature given in Table 3.

8.2 Automatic Distillation Apparatus

For assembly of automatic distillation apparatus, consult the manufacturer's operating manual.

9 CORRECTIONS TO BE APPLIED TO SPECIFIED TEMPERATURES BEFORE COMMENCING THE TEST FOR DISTILLATION YIELD

When the sample has to be tested for distillation yield

Table 3 Condenser Bath Temperature and Sample Temperature
(*Clauses* 8.1.5 *and* 8.1.6)

Sl No.	Initial Boiling Point °C	Condenser Bath Temperature °C	Sample Temperature °C
(1)	(2)	(3)	(4)
i)	Below 50	0 - 3	0 - 3
ii)	50 - 70	0 - 10	10 - 20
iii)	70 - 150	25 - 30	20 - 30
iv)	Above 150	35 - 50	20 - 30

at a specified temperature or in between a specified temperature range the thermometer readings at which the distillate volume in the receiver to be recorded has to be arrived at before starting the distillation process by applying corrections. There are two types of corrections. One is pertaining to Thermometer error and another one pertaining to is Barometric pressure.

9.1 Temperature Adjustments Due to Thermometer Error

If the thermometer gives incorrect readings at the specified distillation temperature as observed during calibration adjust the temperatures by adding the amount of error, if the thermometer is reading low or by subtracting the amount of error if the thermometer is reading high against a known calibration standard.

9.2 Temperature Adjustments Due to Barometric Pressure

When the barometric pressure of location is different from 760 mm of Hg apply adjustments to the specified distillation temperatures as indicated in the specification for the material under test. The quantum of adjustment required can be arrived at using the equation given in **10.2**. This is applicable for atmospheric pressures above 700 mm of Hg.

10 PROCEDURE

10.1 Manual Distillation Procedure

10.1.1 Using the graduated receiver measure 100 ml of the temperature-adjusted sample. Remove the flask from the apparatus and transfer the sample from the graduated receiver into the flask directly. Allow the graduate to drain for 15 to 20 s. Add a few small pieces of clean dry pumice stones or glass beads.

NOTE — For viscous liquids a longer drainage period may be required to complete transfer of the liquid but the drainage time should not exceed 5 min. Do not allow any of the sample to enter the vapour tube.

10.1.2 Connect the flask to the condenser by inserting vapour tube of the flask into the condenser, making a tight connection with a non-reactive cork or ground glass joint which ever is appropriate. Adjust the position of flask support so that the neck of the flask is vertical and the vapour tube extends into the condenser tube as specified in **8.1.4**. Make sure that the flask bottom is resting firmly in the hole of the flask support.

10.1.3 Insert the thermometer into the neck of the flask as described in **8.1.2**.

10.1.4 Place the graduated receiver without drying at the outlet of the condenser tube in such a way that the condenser tube extends into the graduate at least 25 mm, but does not extend below the 100 ml mark.

10.1.5 If the initial boiling point of the sample is below 70°C, immerse the cylinder in a transparent water bath and maintain the temperature of the bath in between 10 and 20°C throughout the distillation.

10.1.6 Place a flat cover on the top of the graduate to prevent condensed moisture entering into the graduate.

10.1.7 Apply heat to the flask and adjust the heating rate so that the first drop of the distillate falls from the condenser tip in to the receiver in between 5 to 10 min from the initial application of heat. For samples having initial boiling point above 150°C this time duration shall be 10 to 15 min.

10.1.8 Observe the temperature on the thermometer as soon as the first drop of the distillate falls in to the graduate and record it as initial boiling point (IBP).

10.1.9 Adjust the heat input after observing initial boiling point so that the distillation proceeds at an uniform rate of 4 to 5 ml/min (approximately 2 drops/s) until 95 percent of the distillate is collected. Move the receiving graduated cylinder in such away that the tip of the condenser touches one side of the cylinder after observing IBP.

10.1.10 Note the volume of the liquid collected in the receiver at the specified, corrected temperature reading on the thermometer for distillation yield.

10.1.11 If distillation yield is not a requirement record the readings of the thermometer after collecting 5, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 95 ml of distillate in to the receiver.

10.1.12 With out changing the heater setting continue distillation beyond 95 percent point, until the dry point is observed (the last drop of liquid evaporates from the bottom of the flask). Record this as dry point.

10.1.13 When dry point could not be observed due to active decomposition (appearance of rapid evolution of heavy fumes) of the liquid, before dry point is reached, observe for the maximum temperature attained on the distillation thermometer. Record this as final boiling point (FBP). For liquids of high boiling range, after attaining the maximum temperature (FBP)

the temperature on the thermometer starts falling for a few degrees.

10.1.14 If the expected drop in temperature does not occur after attaining maximum temperature record the maximum temperature observed on the distillation thermometer 5 min after the 95 percent point has been reached and record this as end point 5 min. In any event the end point does not exceed 5 min after the 95 percent point.

10.1.15 Switch of the heater after the distillation is over.

10.1.16 Allow the flask to cool to room temperature. The vapour in the flask condenses and liquids collects at the bottom of the flask. This is called residue. Drain this liquid carefully into a 10 ml or 5 ml graduated cylinder having 0.1 ml sub-divisions. Measure the volume and record it as residue.

10.1.17 Record the volume of the liquid collected in the receiver after condenser tube has drained completely. This volume is reported as recovery or yield. The recovery shall be 95 percent minimum. If it is less than that repeat the test.

10.1.18 Measure the difference between 100 ml and the sum of recovery plus residue. Record this difference as distillation loss.

11 CORRECTIONS TO BE APPLIED TO THE OBSERVED TEMPERATURES AFTER DISTILLATION (FOR RANGE)

For calculating boiling range of a liquid use the following corrections for observed temperature readings after the distillation is over.

11.1 Corrections for Thermometer Error

If the thermometer gives incorrect reading while calibrating against a calibration standard determine the quantum correction and apply the same for the observed thermometer reading to get correct temperature reading as described in **9.1**.

11.2 Corrections for Barometric Pressure

After applying corrections for thermometer error correct each reading of the distillation for deviation of the barometric pressure. The barometric pressure correction of distillation reading is calculated using the following equation:

$$\text{Correction} = K\,(760 - P)$$

where

K = rate of change in boiling point in °C. This value varies from product to product. K value, in °C, of some of the liquids is given in Table 4.

P = observed barometric reading, in mm Hg, at standard temperature at the location of the test carried out. Correct each reading of distillation by algebraically adding the correction calculated as per the above equation.

If the K value of the liquid under test is not available use the following equation to calculate the approximate K value.

$$K = 0.000\,12(T+273)$$

where

T = observed boiling point in °C.

If the overall distillation range of the sample does not exceed 2°C, the combined correction from thermometer and the barometric pressure variance can

Table 4 *K* Value for Some of the Pure Liquids at Their Boiling Point
(*Clause* 11.2)

Sl No.	Compound Name	*K* Value °C	Boiling Point °C
(1)	(2)	(3)	(4)
i)	Acetone	0.039	56.1
ii)	*n*- Amyl alcohol	0.041	138.0
iii)	*n*- Amyl acetate	0.048	149.5
iv)	Aromatic solvent	0.049	—
v)	Benzene	0.043	80.1
vi)	iso- Butyl Acetate	0.045	117.3
vii)	*n*- Butyl acetate	0.045	126.1
viii)	Sec- Butyl acetate	0.045	112.4
ix)	iso- Butyl alcohol	0.036	107.9
x)	*n*- Butyl alcohol	0.037	117.7
xi)	Sec. Butyl alcohol	0.035	99.5
x)	Diacetone alcohol	0.050	—
xi)	Diethylene glycol	0.050	245.0
xii)	Dipropylene glycol	0.051	232.8
xiii)	Ethyl acetate	0.041	77.2
xix)	Ethyl alcohol	0.033	78.3
xx)	Ethylene glycol	0.045	197.6
xxi)	2- Butoxy ethanol	0.047	171.2
xxii)	2- Ethoxy ethanol	0.044	135.1
xxiii)	2- Ethoxy ethyl acetate	0.046	156.3
xxiv)	Hexylene Glycol	0.045	197.1
xxv)	*n*- Hexyl acetate	0.050	171.6
xxvi)	isophorne	0.057	215.3
xxvii)	Methyl alcohol	0.033	64.5
xxviii)	Methyl Ethyl Ketone	0.043	79.6
xxix)	Methyl Iso amyl acetate	0.048	146.2
xxx)	Methyl Iso amyl ketone	0.048	144.9
xxxi)	Methyl Iso Butyl carbinol	0.041	131.8
xxxii)	Methyl Iso butyl ketone	0.046	116.2
xxxiii)	Perchloroethylene	0.048	121.2
xxxiv)	Isopropyl alcohol	0.033	82.3
xxxv)	Iso propyl acetate	0.041	88.5
xxxvi)	Propylene glycol	0.043	187.6
xxxv)	Pyridine	0.046	115.4
xxxvi)	Toluene	0.046	110.6
xxxvii)	Trichloroethylene	0.043	87.1
xxxviii)	Vinyl acetate	0.040	72.7
xxxix)	Xylene (Mixed isomers)	0.049	—

be estimated by finding the difference between the 50 percent boiling temperature and the true boiling point of the liquid from Table 4.

12 REPORTING

For reporting distillation range, report the corrected initial boiling point and dry point or final boiling point whichever is applicable.

For reporting distillation yield, report the difference between the volumes of distillate recorded as the percentage by volume distilling between the specified temperatures at 760 mm of Hg pressure.

If no definite manner of reporting is specified, report the corrected temperature at each observed volume, and report the volume percentages of the residue, recovery and distillation loss.

ANNEX A
(*Clause* 6.9)

FACTORS CAUSING SUPERHEATING

A-1 In general, any condition whereby the temperature surrounding the vapour exceeds the temperature of the vapour in equilibrium with the liquid will cause superheating. Specific factors conducive to superheating are as follows, and should be avoided.

A-1.1 Flame in Contact with the Flask

The applied gas flame should be prevented from contacting more than the specified portion of the flask by the following procedure:

a) Maintain the correct overall dimensions and specified hole diameter of the asbestos board. The hole must be perfectively circular, with no irregularities;
b) Use a board that is free from cracks and checks; and
c) Set the flask snugly in the hole in the upper insulating board.

A-1.2 Application of Heat

Attention should be given to burner placement, position and character of flame as follows:

a) Apply the source of heat directly beneath the flask. Any variation would result in heating a larger portion of surrounding air to a higher temperature than that of the flask.
b) The flame should not have a larger cross-section than is necessary, and should be non-luminous.
c) Place the burner at a level such that the complete combustion area of a non-luminous flame is approximately 20 mm below the board.

A-1.3 Extraneous Heat Source

An extraneous source of heat such as sunlight falling directly on the flask will cause super heating.

A-1.4 Condition of Equipment

Caution should be observed in employing the apparatus for immediate reuse. For low boiling materials, cool the heating unit to room temperature before starting the test.

A-1.5 Use of Electric Heaters

Electric heaters will, in general, cause superheating. These should used only when after they have been proven to give results comparable to those obtained when using gas heat. The superheating effect obtained from electric heaters may be minimized, but not completely eliminated by selecting a heater which by its design concentrates the heating elements to a minimum area and which contains a minimum amount of ceramic material in its overall construction.

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This Indian Standard has been developed from Doc No.: CHD 1 (1840).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi